# Determination of Available Alumina and Reactive Silica in Bauxite

## Introduction

Bauxite is the main commercial mineral from which aluminium oxide is extracted, which in turn is smelted to form aluminium metal. Two parameters which are often used to assess the viability of a bauxite deposit are described as “Available Alumina” and “Reactive Silica”. Available Alumina defines the net amount of $Al_2O_3$ that can be extracted from the bauxite using conditions that simulate the extraction of alumina in the Bayer process. Reactive Silica is a measure of the amount of silicious materials that will react with the caustic soda in the Bayer process. In low temperature Bayer process refineries, Reactive Silica is primarily sourced from the dissolution of clay minerals, however as processing temperatures increase, attack on quartz also contributes to the Reactive Silica component. Reactive Silica is an important parameter of a bauxite deposit because the silica that goes into solution is subsequently precipitated as a form of sodium aluminium silicate which results in significant cost because of consumption of caustic soda.

Traditionally, laboratory scale stainless steel high pressure vessels have been used to carry out these digests, commonly at 145 °C but up to 250 °C to simulate conditions in some processing plants. Difficulties have always been experienced with this equipment in controlled pre-heating of the vessels to working temperature and then maintenance of the digestion temperature for the holding time of (typically) 30 minutes.

Control of these parameters in microwave heated vessels seemed to offer an elegant and more efficient way of controlling this process.

One critical parameter for the success of a microwave digestion process is that there must be no vapor phase leakage from the vessels. If leakage occurs, the caustic soda concentration would rise and erroneous results would result. In contrast to some other makes and with proper maintenance, the Anton Paar vessels were found to be leak free and successful analyses were able to be made. To confirm whether leakage had occurred, vessel assemblies were weighed before and after the digestion process.

Because there are a wide range of digestion parameters specified by bauxite refineries, there are no certified reference materials that cover all nominated digestion conditions. The purpose of this evaluation therefore was:

1. To determine analytical precision under a specific set of digestion conditions.
2. To monitor possibility of vessel leakage during digestion.
3. In the case of the 48 position rotor, to determine if there was any systematic variability between outer, middle and inner circle of vessels.

## Instrumentation

Digestions were carried out with a Multiwave 3000 oven equipped with a 48 position rotor (48MF50) made by **Anton Paar** GmbH, Graz. Digestion solutions have been analysed by a number of different ICP-AES instruments and it appears that this part of the analytical procedure is not manufacturer specific.

## Samples

Three bauxite samples were selected that covered a range of concentrations of both Available Alumina and Reactive Silica. The material was sourced from bauxite deposits in the South West of Western Australia. Digestion conditions primarily target the mineral gibbsite in the ore.

## Analytical procedure

1. 2.00 grams sample are weighed into the vessels.
2. 10.0 mLs of caustic soda (145 g/L) is added to the vessel.
3. A stir bar is added.
4. Vessels are closed and then weighed.

5. The loaded rotor is placed in the oven and the following digestion profile is used.

Program:

| | Temp [°C] | Ramp [min] | Hold [min] | Fan |
|---|---|---|---|---|
| 1 | 175 | 30:00 | 30:00 | 1 |
| 2 | 0 | 0 | 15:00 | 3 |

Stirrer setting 2 is used through the whole process.

6. After digestion the vessels are again weighed to monitor any weight loss caused by vessel leakage. The vessel contents are diluted to 500 mL with water and mixed. A 10 mL portion is removed for determination of aluminium by direct aspiration to an ICP-AES instrument. Available Alumina is expressed as a percentage of $Al_2O_3$ in the sample.
7. The remaining solution (from the 500 mL diluent) is then acidified with 10.0 mLs of hydrochloric acid and mixed. Acidification re-dissolves the sodium aluminium silicate that has precipitated. This solution is sub-sampled and silicon concentration is determined by direct aspiration into an ICP-AES instrument. Reactive Silica is expressed as a percentage of $SiO_2$ in the sample.
8. For both ICP determinations, the instrument is calibrated against synthetically prepared standard solutions. Calibration solutions for Available Alumina range from the equivalent of 10% Available Alumina ($Al_2O_3$) in the sample through to 60% and are prepared in a matrix equivalent to the sample solutions.

Calibration solutions for Reactive Silica range from the equivalent of 1% Reactive Silica ($SiO_2$) in the sample through to 30% and are also prepared in a matrix equivalent to the samples.

## Results

Each sample has been analysed 5 times in the inner, middle and outer circle of the rotor. In all cases, the loss in weight before and after digestion was less than 0.1 grams indicating there was no significant leakage in all of these tests.

| | Available Aluminia % | | | Reactive Silica % | | |
|---|---|---|---|---|---|---|
| Sample | Inner | Middle | Outer | Inner | Middle | Outer |
| 1-1 | 31.4 | 31.0 | 31.0 | 1.4 | 1.7 | 1.4 |
| 1-2 | 31.2 | 31.2 | 31.2 | 1.5 | 1.3 | 1.5 |
| 1-3 | 31.2 | 31.0 | 31.2 | 1.6 | 1.3 | 1.5 |
| 1-4 | 30.8 | 31.4 | 31.2 | 1.8 | 1.5 | 1.7 |
| 1-5 | 31.4 | 31.4 | 30.8 | 1.6 | 1.5 | 1.6 |
| Average | 31.2 | 31.2 | 31.1 | 1.6 | 1.5 | 1.5 |
| 2-1 | 27.8 | 28.1 | 29.3 | 1.8 | 1.7 | 1.9 |
| 2-2 | 27.8 | 27.4 | 28.1 | 1.7 | 1.7 | 1.6 |
| 2-3 | 27.8 | 28.0 | 28.1 | 1.7 | 1.6 | 1.7 |
| 2-4 | 28.3 | 27.4 | 27.8 | 1.8 | 1.6 | 1.7 |
| 2-5 | 28.1 | 27.0 | 28.7 | 1.8 | 1.6 | 1.8 |
| Average | 28.0 | 27.6 | 28.4 | 1.8 | 1.6 | 1.7 |
| 3-1 | 25.1 | 24.7 | 24.7 | 5.7 | 5.7 | 5.8 |
| 3-2 | 24.9 | 24.9 | 24.2 | 5.6 | 5.7 | 5.9 |
| 3-3 | 25.1 | 24.9 | 24.6 | 5.8 | 5.7 | 5.8 |
| 3-4 | 24.7 | 24.6 | 24.2 | 5.8 | 5.7 | 5.9 |
| 3-5 | 24.9 | 24.4 | 24.6 | 5.8 | 5.7 | 5.8 |
| Average | 24.9 | 24.7 | 24.5 | 5.7 | 5.7 | 5.8 |

## Conclusion

Microwave heating of samples for this digestion process has proved to be highly reproducible and excellent analytical precision has been achieved. The Anton Paar vessels have been found to be easy to use and they do not leak when properly used and maintained. Although the reported data is a relatively small data set, there does not appear to be any significant difference in heating or other digestion conditions between the outer, middle and inner circle of the 48 position rotor.

## References

Ultra Trace Analytical Laboratories, Australia